

PCTWORLD INTELLECTUAL PROPERTY ORGANIZATION
International Bureau

INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification ⁵ : A61K 7/00, 7/027	A1	(11) International Publication Number: WO 94/06400 (43) International Publication Date: 31 March 1994 (31.03.94)
(21) International Application Number: PCT/US93/08684 (22) International Filing Date: 15 September 1993 (15.09.93) (30) Priority data: 07/947,692 21 September 1992 (21.09.92) US 08/066,351 21 May 1993 (21.05.93) US 08/115,093 7 September 1993 (07.09.93) US (71) Applicant: THE PROCTER & GAMBLE COMPANY [US/US]; One Procter & Gamble Plaza, Cincinnati, OH 45202 (US). (72) Inventors: EL-NOKALY, Magda ; 2357 Bedford Avenue, Cincinnati, OH 45208 (US). WALLING, David, William; 2 Andrews Court, Parkton, MD 21120 (US). VATTER, Michael, Lee ; 4159 California Road, Okeana, OH 45053 (US). LEATHERBURY, Neil, Campbell ; 519 W. Pratt Street, #309, Baltimore, MD 21201 (US).		(74) Agents: REED, T., David et al.; The Procter & Gamble Company, 5299 Spring Grove Avenue, Cincinnati, OH 45217 (US). (81) Designated States: AU, CA, JP, KR, European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE). Published <i>With international search report.</i>
(54) Title: LIPSTICKS (57) Abstract The present invention relates to lipstick compositions which comprise thermodynamically association structures, preferably lamellar liquid crystals and/or reverse hexagonal liquid crystals, which are used to deliver polar solvent/moisturizers through a non-polar (lipophilic) medium. The association structures consist essentially of from about 3 % to about 96 % by weight, of polar solvent (e.g. glycerine) and from about 4 % to about 97 % of surfactant having a Krafft point at or below about ambient temperature.		

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AT	Austria	FR	France	MR	Mauritania
AU	Australia	GA	Gabon	MW	Malawi
BB	Barbados	GB	United Kingdom	NE	Niger
BE	Belgium	GN	Guinea	NL	Netherlands
BF	Burkina Faso	GR	Greece	NO	Norway
BG	Bulgaria	HU	Hungary	NZ	New Zealand
BJ	Benin	IE	Ireland	PL	Poland
BR	Brazil	IT	Italy	PT	Portugal
BY	Belarus	JP	Japan	RO	Romania
CA	Canada	KP	Democratic People's Republic of Korea	RU	Russian Federation
CF	Central African Republic	KR	Republic of Korea	SD	Sudan
CG	Congo	KZ	Kazakhstan	SE	Sweden
CH	Switzerland	LI	Liechtenstein	SI	Slovenia
CI	Côte d'Ivoire	LK	Sri Lanka	SK	Slovak Republic
CM	Cameroon	LU	Luxembourg	SN	Senegal
CN	China	LV	Latvia	TD	Chad
CS	Czechoslovakia	MC	Monaco	TC	Togo
CZ	Czech Republic	MG	Madagascar	UA	Ukraine
DE	Germany	ML	Mali	US	United States of America
DK	Denmark	MN	Mongolia	UZ	Uzbekistan
ES	Spain			VN	Viet Nam
FI	Finland				

LIPSTICKS

5

FIELD OF THE INVENTION

10 This invention relates to lipstick compositions which comprise thermodynamically stable association structures which are used to deliver polar moisturizing agents through a nonpolar (lipophilic) matrix. The invention provides for a lipstick with optimum stability of the polar moisturizing agents, while also providing moisturizing benefits to the lips.

15

BACKGROUND OF THE INVENTION

Lipsticks are primarily composed of hydrocarbon waxes, esters of fatty acids or alcohols and oils. Water and other polar solvents, such as glycerine and propylene glycol, which can provide moisturization benefits are not soluble in this lipophilic matrix. As a result, various emulsifiers have been added to lipsticks in an attempt to emulsify the moisturizers/polar solvents. There has been partial success in this area by utilizing the emulsifiers and moisturizers/polar solvents at low concentrations. However, the moisturizers/polar solvents may separate from the lipstick causing it to look wet and messy. This is unacceptable to the consumer.

25 Thus a need exists to provide a lipstick which binds thermodynamically the moisturizers/polar solvents (discontinuous phase) and deliver them in a predominately nonpolar lipophilic matrix (continuous phase). Further, there is a need to provide a means of thermodynamically binding the moisturizers/polar solvents in such a way which will allow incorporation of high levels of the moisturizing agents while exhibiting overall excellent stability and providing good feel properties.

30 It has been discovered that association structures consisting essentially of a surfactant or mixture of surfactants having a Krafft point at or below about ambient temperature (about 20°C) and a moisturizer/polar solvent can thermodynamically bind the moisturizer/polar solvent and homogeneously absorb in the lipophilic matrix while providing good feel and a means of delivering the moisturizing agents to the lips. Thus, the association structures of this invention can be used to deliver the moisturizers/polar solvents without syneresis, the separation of the hydrophilic materials. It has been discovered that upon application to the lips the preferred association structures of the present invention form a film on the lips which can act as a reservoir for the moisturizer/polar solvent.

It is particularly desirable that the lipsticks comprising said association structures be made from ingredients that are presently used and approved for use in edible product applications and for applying to the skin. It is an object of this invention to provide a delivery system for moisturizers/polar solvents through the nonpolar matrix of the lipstick while providing good feel. It is another object of this invention to provide a lipstick composition which can be easily molded yet contain high levels of moisturizers.

Sweating, the excretion of oil on the surface of a lipstick, is a common problem. Lipsticks are a complex mixture of solid, semi-solid and liquid lipids. Wax networks suspend the oils present in the lipstick. Sweating occurs due to the inferior oil-binding capacity of the wax network and/or to a high oil content causing supersaturation. The phenomenon can occur in any climate or temperature; although, it is more apt to occur in sub-tropical and tropical climates.

In most cases, castor oil has been identified as the excreted oil. A major ingredient of most lipstick formulations, castor oil can generally comprise up to forty percent of lipsticks. The high percentage of castor oil is primarily due to its unique properties as a wetting agent for pigments. Monodispersions of pigments are commonly made in slurries comprising from about 30% to about 40% pigments and from about 60% to about 70% castor oil.

It is an object of this invention to provide a preferred lipstick composition of the present invention which is substantially free of castor oil. It is an object that the lipstick be castor oil sweat resistant or castor oil sweat free. An added benefit to removing the castor oil is the removal of the castor oil odor.

It has been discovered that the association structures, preferably lamellar liquid crystals (neat phase), can be used to deliver moisturizing agents in lipsticks which are substantially free of castor oil. It has been found that removal of the castor oil, typically present in the range of from about 30% to about 50% of the lipstick compositions, allows for optimization of emollients; thus, providing for a more consumer acceptable feel, such as less tackiness, and moisturizing benefits by utilizing more lubricious emollients.

All percentages and ratios stated herein are by weight unless otherwise indicated.

SUMMARY OF THE INVENTION

The present invention relates to lipstick compositions which comprise association structures which are used to deliver moisturizing agents/polar solvents into a non-polar/lipophilic medium in a thermodynamically stable way. A method of making the lipstick is also disclosed.

The lipstick compositions comprise:

- (a) from about 5% to about 90%, by weight, of wax;

- (b) from about 1% to about 90%, by weight, of an emollient component comprising from 0% to about 100%, by weight, of oil liquid at ambient temperature;
- (c) from about 0.1 to about 80%, by weight, of an association structure consisting essentially of:
- 5 (1) from about 3% to about 96%, by weight, of polar solvent; and
- (2) from about 4% to about 97% by weight, of surfactant having a Krafft point at or below about ambient temperature; and
- (d) from 0% to about 35%, on an anhydrous basis, of color;
- 10 Preferred lipstick compositions are substantially free of castor oil, contain a coupling agent, and/or have compatible solubilities for wax and oil components. These lipstick compositions provide moisturizing, long wear and good feel properties.

Without intending to necessarily limit the scope of the invention, it is believed that the association structures adsorb onto solid/liquid, or liquid/liquid interfaces in

15 heterogeneous systems such as lipsticks and other cosmetic sticks. An association structure, preferably a liquid crystalline lamellar phase, forms in the continuous phase. These association structures are able to flow under shear and act as lubricants between the different components of the heterogeneous systems such as the solids and other materials in the product. They also stabilize the actives by entrapping the liquid, solids

20 particles or droplets in their matrix and preventing them from flocculating and further coalescing.

DETAILED DESCRIPTION OF THE INVENTION

Definition

As used herein, the term "solid material" refers to any solid lipstick ingredient

25 capable of adsorbing the association structures. Solids include waxes, solid fats, waxy emulsifiers or pigments commonly used in lipsticks.

As used herein, "color(s)" includes pigments, dyes, colors, lakes, and pearl. Colors are measured on an anhydrous weight basis.

As used herein, the term "lecithin" refers to a material which is a phosphatide.

30 Naturally occurring or synthetic phosphatides can be used. Phosphatidylcholine or lecithin is a glycerine esterified with a choline ester of phosphoric acid and two fatty acids, usually a long chain saturated or unsaturated fatty acid, having 16-20 carbons and up to 4 double bonds. Other phosphatides capable of forming association structures, preferably lamellar or hexagonal liquid crystals, can be used in place of the lecithin or in

35 combination with it. Other phosphatides are glycerol esters with two fatty acids as in the lecithin, but the choline is replaced by ethanolamine (a cephalin), or serine (a-aminopropanoic acid; phosphatidyl serine) or an inositol (phosphatidyl inositol).

As used herein, the term "surfactant" refers to a low molecular weight or monomer non-polymeric organic compound amphiphilic in nature, i.e., it has hydrophilic and hydrophobic groups and exhibits a marked tendency to adsorb on a surface or interface and lower the surface tension. Surfactants or emulsifiers are divided into nonionic (no charge), anionic (negative charge), cationic (positive charge) and amphoteric (both charges) based on whether or not they ionize in aqueous media. Surfactants are monomers and are derived from natural oils and fats and crude oils. The term "surfactant" as used herein refers to mixtures of surfactants as well as a single organic compound.

As used herein, "polar solvent" means a polar material capable of forming an association structure with a surfactant. Some examples of polar solvents include glycerine, panthenol (preferably panthenol mixed with glycerine or alcohol), propylene glycol, butylene glycol, water, alcohols, alkanediols, polyethylene glycols, sorbiton, maltitol and mixtures thereof.

As used herein the term "comprising" means that the composition can contain other ingredients which are compatible with the composition and which preferably do not substantially disrupt the association structure lipstick compositions of the present invention. The term encompasses the terms "consisting of" and "consisting essentially of".

Essential Components Association Structures

As used herein "association structure" refers to reverse micelle and lyotropic liquid crystal structures which are formed by the mixture of a surfactant or mixture of surfactants and a polar solvent or mixture of polar solvents at ambient temperature. The liquid crystalline state is an intermediate state between the solid and liquid states. It is often called a mesomorphic state. The association structures of the present invention are thermodynamically stable. They are distinguishable from gels or emulsions which have the polar solvent separate when subjected to ultracentrifugation. Separation means that generally at least 50%, preferably at least 80% and more preferably at least 99%, of the polar solvent separates. Further, they are distinguished in that the lyotropic liquid crystalline state can be identified by one of ordinary skill in the art by known means such as transmission electron microscopy (TEM) or birefringence under polarized light and x-ray diffraction.

In the literature, association structures are also referred to as anisotropic fluids or in the case of the cubic phase as isotropic fluids, a fourth state of matter, liquid crystals, aggregates, or mesophases. These terms are used interchangeably. Association structures or aggregates are generally disclosed in the reference Lyotropic Liquid Crystals Stig Friberg (Ed.), American Chemical Society, Washington, D.C., 1976, pp 13-27 which is herein incorporated by reference.

The association structures of the present invention, are prepared by mixing a surfactant having a Krafft point at or below about ambient temperature with a sufficient amount of a polar solvent to form the desired association structure. Each surfactant has a temperature and concentration range in which the association structure will exist based on the surfactant's chemical structure, the type of solvent being used, and the presence of any impurities. The liquid crystalline phase flows under shear and is characterized by a viscosity that is significantly different from the viscosity of its isotropic solution phase. Rigid gels do not flow under shear like liquid crystals. Also, when viewed with a polarized light microscope, liquid crystals show identifiable birefringence, as, for example, planar lamellar birefringence, whereas when isotropic solutions and rigid gels are viewed under polarized light, both show dark fields. Exceptions to this method of detection can occur for example with the cubic phases which can not be detected by a polarized light microscope but can be detected by x-ray diffraction. Other methods of detection commonly used by ones of ordinary skill in the art are given infra.

Adding a gel or emulsion of a surfactant with a polar solvent to a fat, oil, wax or other hydrophobic medium often leads to unacceptable results because they are not thermodynamically stable. Emulsifying the oil/water and surfactant does not provide a thermodynamically stable system. The polar solvent would be expected to separate during storage or use and with changes in temperature. Adding the association structures of the present invention to the same system provides a system which is stable on storage because the association structure of the surfactant and polar solvent are thermodynamically stable and adsorb on the wax. The association structures can tolerate wide changes of temperatures, e.g. from ambient temperature to about 100°C. The polar solvent is bound within multilayers and does not separate, even when ultracentrifuged.

Micelles are large polymolecular aggregates in solutions. Normal micelles predominate in surfactant solutions above the critical micelle concentration which occurs at the Krafft temperature. The lipophilic groups accumulate in the liquid-like inner part of the aggregates. The hydrophilic groups are directed out towards the water. "Inverted" micelles in a hydrocarbon environment have their polar groups piled up in the inner part of the micelles. These reverse micelles can aggregate to form spherical, elongated, cylindrical, filament structures or mixtures thereof which can network in the hydrocarbon environment. The term "reverse micelles", as used herein, refers to these aggregates of reversed micelles which are the spherical, elongated, cylindrical, or filament structures and/or mixtures thereof. The spherical are liquid-like and as they become larger, i. e., elongated, they are gel-like.

One type of association structure, the liquid crystals, are a fourth state of matter. They exist between the boundaries of the solid phase and the isotropic liquid phase (i.e. an intermediate between the three dimensionally ordered crystalline state and the disordered dissolved state). In this state some of the molecular order characteristics of the solid phase are retained in the liquid state because of the molecular association structure and long range intermolecular interaction. The ability of some compounds to form a mesophase, typically referred to as liquid crystals, was observed nearly a century ago.

Thermotropic liquid crystals are obtained by heating solid crystals at a temperature above which they are no longer stable. Such thermotropic liquid crystals are well known in our day-to-day life, and have multiple applications as they exhibit variations in color with temperature and/or a magnetic field and/or an electric field. They are formed by elongated molecules and are used in some cosmetics for their visible impact (visualization of actives). Lyotropic liquid crystals result from the interaction with a solvent over a particular range of concentration and temperature. Low molecular weight lyotropic liquid crystals, i.e. liquid crystals formed from a low molecular weight emulsifier or organic amphiphile (a compound having both a polar and a nonpolar group, as a soap, lecithins or long chain fatty acid monoglyceride), are known to encapsulate and act as a delivery vehicle for drugs, flavors, nutrients and other compounds.

The association structures of the present invention are:

a) Reverse Micelles:

- (1) Reverse micelles also known in the art as spherical reverse micelles, elongated reverse micelles, bicontinuous phase or L2 phase; and
- (2) Cylindrical reverse micelles or reverse connected rod-shaped liquid crystals also known in the art as networking reverse cylinders, connected cylindrical reverse micelle structures, or connected cylinders; and

b) Lyotropic Liquid Crystals:

- (1) Reverse hexagonal liquid crystals also known in the art as Hexagonal II or F phase;
- (2) Cubic liquid crystals also known in the art as viscous isotropic and I₂ phase; and
- (3) Lamellar liquid crystals also known in the art as the La neat phase and D phase.

The association structure of the present invention is selected from the group consisting of reverse micelles, lyotropic liquid crystals and mixtures thereof

Preferred association structures are the cylindrical reverse micelle, reverse hexagonal liquid crystals, cubic liquid crystals, lamellar liquid crystals and mixtures thereof. The most preferred association structures are lamellar liquid crystals, reverse hexagonal liquid crystals and mixtures thereof. The association structures can be in the following phases: two phase liquid crystals, one phase liquid crystals, reverse micelles/liquid crystalline phase or liquid crystalline/solvent phase. Preferably the liquid crystals are substantially one phase or two liquid crystalline phases, i.e., at least about 90%, more preferably about 98% and most preferably at least about 99%, of the association structure is in the form of the liquid crystal.

The association structures comprise from about 0.1% to about 80% of the lipstick composition. Preferably the association structures comprise from about 3% to about 75%, more preferably from about 10% to about 65%, and most preferably from about 30% to about 60% of the lipstick composition comprises the association structures, preferably lamellar liquid crystals.

Polar Solvents

The solvents useful for making the association structures of the present invention include any polar solvent acceptable for human ingestion. Suitable polar solvents include: water, alcohols, such as ethanol, propyl alcohol, isopropyl alcohol, hexanol, and benzyl alcohol; polyols, such as propylene glycol, polypropylene glycol, butylene glycol, maltitol, sorbitol, and glycerine; panthenol dissolved in glycerine; flavor oils, and mixtures thereof. Mixtures of these solvents can also be used. Preferred polar solvents are glycerine, panthenol in glycerine, propylene glycol, butylene glycol, water and mixtures thereof. Most preferably, water added by itself, i.e. other than the water present in commercially supplied solvents, is not utilized. Thus, the most preferred lipstick compositions of the present invention are essentially free of water, i.e., less than about 3% and preferably less than about 1%. The most preferred polar solvents for use are glycerine, panthenol, propylene glycol, butylene glycol and mixtures thereof.

The solvents are used at levels of from about 3% to about 96%, preferably from about 5% to about 95%, more preferably from about 10% to about 80% and most preferably from about 30% to about 70% of the association structure. Typically, the lipstick compositions will comprise from about 0.1% to about 60%, preferably from about 1% to about 30% and most preferably from about 8% to about 18% polar solvent.

Surfactants

Surfactants suitable for use are those which can form association structures, preferably lamellar liquid crystals or reverse hexagonal, at ambient temperature when mixed with a polar solvent. Ambient temperature/room temperature as used herein typically means about 20°C. Generally ambient temperature can range from about 18°C to about 27°C, preferably from about 20°C to about 25°C, depending on such variables as geographical location, i.e. sub-tropical vs. temperate regions. One of ordinary skill in

the art is able to determine if association structures form at ambient temperatures. The surfactants suitable for use generally have a Krafft point at or below about ambient temperature about 20°C or generally at or below about 18°C to about 27°C, preferably at or below from about 20°C to about 25°C.

- 5 The definition of Krafft point is well known in the art and one of ordinary skill in the art can determine a surfactant's Krafft point. In general terms, Krafft point is the melting point of the hydrocarbon chains of the surfactants. It can also be expressed as the temperature at which the solubility of an association colloid in water suddenly increases because critical micelle concentration is exceeded and micelles form. See
10 Ekwall., P., "Composition, Properties and Structure of Liquid Crystalline IPhases in Systems of Amphiphilic Compounds" Advances in Liquid Crystals Vol. I, Chapter I, p.81.

- In preparing a sample combination of surfactant and polar solvent to demonstrate the ability to form association structures, the surfactant needs to be sufficiently soluble in
15 the polar solvent such that an association structure can form at ambient temperature. One of ordinary skill in the art is capable of determining compatible interactions.

- Any surfactant which forms association structures at ambient temperature and is suitable for use in cosmetics is suitable for use herein. Surfactants suitable for use in cosmetics do not present dermatological or toxicological problems. Anionic surfactants,
20 nonionic surfactants, cationic surfactants, amphoteric surfactants and mixtures thereof are suitable for use. Preferably anionic surfactants, nonionic surfactants, cationic surfactants, amphoteric surfactants and mixtures thereof having a Krafft point at or below about ambient temperature are used. More preferably, nonionic surfactants, cationic surfactants, amphoteric surfactants and mixtures thereof having a Krafft point at
25 or below about ambient temperature are used.

- Types of anionic surfactants suitable for use are soaps; sulfonates such as alkane sulfonates (e.g., branched sodium x-alkane sulfonate where $x \neq 1$) paraffin sulfonates, alkylbenzene sulfonates, α -olefin sulfonates, sulfosuccinates and sulfosuccinate esters (e.g., dioctylsodium and disodium laureth sulfosuccinate), oisethionates,
30 acylisethionates (e.g., sodium 2-lauroyloxyethane sulfonate), and sulfalkylamides of fatty acids, particularly N-acylmethyltaurides; sulfates such as alkyl sulfates, ethoxylated alkyl sulfates, sulfated monoglycerides, sulfated monoglycerides, sulfated alkanolamides, and sulfated oils and fats; carboxylates such as alkyl caboxylate having a carbon chain length above C₁₂, acylsarcosinates, sarcosinates (e.g., sodium lauryl sarcosinate), ethoxylated
35 carboxylic acid sodium salts, carboxylic acids and salts (e.g., potassium oleate and potassium laurate), ether carboxylic acids; ethoxylated carboxylic acids and salts (e.g., sodium carboxymethyl alkyl ethoxylate; phosphoric acid esters and salts (e.g., lecithin); acylglutamates (e.g., disodium n-lauroyl glutamate) and mixtures thereof. It should be noted that the safest alkyl sulfates for use generally have a hydrocarbon chain lengths

above C₁₂.

Types of nonionic surfactants suitable for use are polyoxyethylenes such as ethoxylated fatty alcohols, ethoxylated alcohols (e.g., octaoxyethelene glycol mono hexadecyl ether, C₁₆E₈ and C₁₂E₈), ethoxylated fatty acids, ethoxylated fatty amines, 5 ethoxylated fatty amides, ethoxylated alkanolamides, and ethoxylated alkyl phenols; triesters of phosphoric acid (e.g., sodium dioleoylphosphate); alkyl amido diethylamines; alkylamido propylbetaines (e.g., cocoamido propylbetaine); amine oxide derivatives such as alkyl dimethylamine oxides, alkyl dihydroxyethylamine oxides, alkyl amidodimethylamine 10 oxides and alkyl amidodihydroxyethylamine oxides; polyhydroxy derivatives such as polyhydric alcohol esters and ethers (e.g., sucrose monooleate, cetostearyl glucoside, β octyl glucofuranoside, esters, alkyl glucosides having a carbon chain length of from C₁₀ to C₁₆), mono, di- and polyglycerol ethers and polyglycerol esters (e.g., tetraglycerol monolaurate and monoglycerides, triglycerol monooleate (such as TS-T122 supplied by Grinsted), diglycerol monooleate (such as TST-T101 supplied by Grinsted), ethoxylated 15 glycerides; monoglycerides such as monoolein and monlinolein; diglyceride fatty acids such as diglycerol monoisostearate (e.g., Cosmol 41 fractionated supplied by Nisshin oil Mills, Ltd.) and mixtures thereof.

Types of cationic surfactants suitable for use are aliphatic-aromatic quaternary ammonium halides; quaternary ammonium alkyl amido derivatives; alkyl 20 amidopropyl dimethylammonium lactate; alkylamidopropyl dihydroxyethylammonium lactate; alkyl amidopropyl morpholinium lactate; quaternary ammonium lanolin salts; alkyl pyridinium halides; alkyl isoquinolinium halides; alkyl isoquinolinium halides; quaternary ammonium imidazolinium halides; bisquaternary ammonium derivatives; alkylbenzyl dimethylammonium salts such as stearylalkylammonium chloride; alkylbetaines 25 such as dodecyldimethylammonium acetate and oleyl betaine; alkylethylmorpholinium ethosulfates; tetra alkyl ammonium salts such as dimethyl distearyl quaternary ammonium chloride and bis isostearamideopropyl hydroxypropyl diammonium chloride (Schercoquat 2IAP from Scher Chemicals); heterocyclic ammonium salts; bis(triacetylammoniumacetyl)diamines and mixtures thereof.

30 Types of amphoteric surfactants suitable for use are alkyl betaines; alkanolamides such as monoalkanolamides and dialkanolamides; alkyl amido propylbetaines; alkyl amidopropylhydroxysultaines; acylmonocarboxy hydroxyethyl glycinate; acyldicarboxy hydroxyethyl glycinate; alkyl aminopropionates such as sodium laurimino dipropionate; alkyl iminodipropionates; amine oxides; acyl ethylenediamine betaines; N-alkylamino 35 acids such as sodium N-alkylamino acetate; N-lauroylglutamic acid cholesterol esters; alkyl imidazolines and mixtures thereof.

Preferred anionic surfactants for use are sulfosuccinate esters, isethionates, sarcosinates, sodium lauryl sulfoacetate, phosphate esters, alkyl carboxylates having a hydrocarbon chain length above C₁₂, acylglutamates and mixtures thereof.

Most preferred for use are nonionic surfactants. Examples of preferred nonionic surfactants are carbohydrate surfactants such as sucrose monoester and alkyl glucosides; polyglycerol esters such as tetraglycerol monolaurate PG-3 diisostearate, triglycerol monooleate, and diglycerol monooleate; monoglycerides; diglycerol esters such as PG-2 monoisostearate, PG-2 monooleate, PG-2 monostearate, PG2 diisostearate, and PG-2 dioleate; sorbitan esters and mixtures thereof.

Preferred surfactants for use are polyhydric alcohol esters and ethers such as sucrose monooleate, cetoarylglucoside, cetostearylglucoside, alkylglucosides having a carbon chain length of from C₁₀ to C₁₆, β octyl glucofuranosides; polyglycerol esters such as tetraglycerol monooleate or laurate; monoglycerides such as monoolein; phosphatides such as lecithin; bis isostearamidopropyl hydroxypropyl diammonium chloride; sorbitan oleate; dipentaerythritol fatty acid ester; n-lauroyl glutamic acid ester; tetra glycerol monolaurate; and mixtures thereof.

A variety of lecithins can be used. American Lecithin Company (Danbury, CT) supplies a Nattermann Phospholipid, Phospholipon 80 and Phosal 75. All of these function well in this system. Other lecithins which can be used alone or in combination with these are: hydrogenated lecithin supplied by Nisshin Oil Mills, Ltd; Actiflo Series, Centrocap series, Central Ca, Centrol series, Centrolene, Centrolex, Centromix, Centrophase and Centrolphil Series from Central Soya (Ft. Wayne, IN); Alcolec and Alcolec 439-C from American Lecithin; Canasperse from Canada Packers, Lexin K and Natipide from American Lecithin; and L-Clearate, Clearate LV and Clearate WD from the W. A. Cleary Co. Lecithins are supplied dissolved in ethanol, fatty acids, triglycerides and other solvents. They are usually mixtures of lecithins and range from 15% to 75% of the solution as supplied. The lecithins are also supplied as powders. The purity of the powder varies, but the lecithin can be from 60% to 90% of the powder on a weight basis. The weight of phosphatide as used herein is the weight of the lecithin and not of the carriers or impurities.

In order to form the appropriate type of association structure, the lecithin must be sufficiently soluble in the polar solvent such that a liquid crystalline state can be formed at the temperature conditions of product preparation. Additionally, the lecithin association structures should be of a type which has the capability to flow under application of shear, preferably lamellar, hexagonal II (reverse hexagonal) or mixtures thereof.

Both natural and synthetic lecithins can be used. Natural lecithins are derived from oilseeds such as sunflower seeds, soybeans, safflower seeds and cottonseed. The lecithins are separated from the oil during the refining process. Eggs are also a natural source of lecithin.

The phosphatide can be used at a level of from about 25% to about 95%, preferably from about 30% to about 85% and most preferably from about 40% to about

70% of the association structure. Preferably a mixture of a phosphatide with other surfactants capable of forming associations structures is used. When such a mixture is used the phosphatide is preferably used at levels of from about 0.1% to about 30%, preferably from about 0.1% to about 5% and more preferably from about 0.1% to about 1% of the lipstick composition. Most preferably lecithin is not utilized as an association structure forming surfactant, i.e., essentially free of lecithin (>.01%).

Typically when utilizing a phosphatide as the surfactant for forming an association structure at levels of less than about 30% of the association structure, reverse micelles, cylindrical reverse micelles, reverse connected rod-shaped liquid crystals, and mixtures of these association structures will be formed. Typically when utilizing a phosphatide at greater than about 30% of the association structure, the preferred lamellar (L2) phase association structures will be formed.

Typical Formulations Can Utilize the Following:

Amphoteric

- N-alkyl amino acids (e.g., sodium N-alkylaminoacetate)
- Δ • N-lauroylglutamic acid cholesterol ester (e.g., Eldew CL-301 Ajinomoto)

Anionic

- Acylglutamates (e.g., disodium N-lauroylglutamate)
- Δ • Sarcosinates (e.g., sodium lauryl sarcosinate) (Grace, Seppic)
- Taurates (e.g., sodium lauryl taurate, sodium methyl cocoyl taurate)
- Δ • Carboxylic acids and salts (e.g., potassium oleate, potassium laurate, potassium-10-undecenoate; potassium, 11-p-Styryl) - undecanoate
- Ethoxylated carboxylic salts (e.g., sodium carboxy methyalkyl ethoxylate)
- Ether carboxylic acids
- Δ • Phosphoric acid esters and salts (e.g., lecithin) DEA-oleth-10 phosphate
- Acyl isethionates such as sodium 2-lauroyloxyethane sulfonate
- Alkane sulfonates (e.g., branched sodium x-alkane sulfonate (x/1)
- Sulfosuccinates e.g. dioctyl sodium sulfosuccinate; disodium laureth sulfosuccinate (MacKanate El, McIntyre Group Ltd.)
- Sulfosuccinates (aerosols)
 - Sodium dibutyl sulfosuccinate
 - Sodium Di-2-pentyl sulfosuccinate
 - Sodium Di-2-ethylbutyl sulfosuccinate
 - Sodium Di hexyl sulfosuccinate
 - Sodium Di-2 ethylhexyl sulfosuccinate (AOT)
 - Sodium Di-2-ethyldodecyl sulfosuccinate
 - Sodium Di-2-ethyloctadecyl sulfosuccinate
- Sulfuric acid esters, e.g., sodium 2-ethylhept-6-ENYL sulfate; sodium 11-Heneicosyl sulfate; sodium 9-Heptadecyl sulfate
- Alkyl sulfates e.g., MEA alkyl sulfate such as MEA-lauryl sulfate

Cationic

- Alkyl Imidazolines such as alkyl hydroxyethyl imidazoline, stearyl hydroxyethyl imidazoline (supplier Akzo, Finetex and Hoechst)
- Ethoxylated Amines such as PEG-n alkylamine, PEG-n alkylamino propylamine, Poloxamine e.g., PEG-cocopolyamine, PEG-15 tallow amine
- 5 • Quaternaries: Alkylbenzyl dimethyl ammonium salts, betaines, heterocyclic ammonium salts and tetra alkylammonium salts.
Alkylamines, dimethyl alkylamine, dihydroxyethyl alkylamine dioleate
- Alkylbenzyl dimethylammonium salts (e.g., stearyl ammonium chloride)
- Alkyl betaines (e.g., dodecyl dimethyl ammonio acetate, oleyl betaine)
- 10 • Alkyl ethyl morpholinium Ethosulfate
- Tetra alkyl ammonium salts (e.g., dimethyl distearyl quaternary ammonium chloride (Witco))
- Δ • Bis isostearamidopropyl hydroxy propyl diammonium chloride (Schercoquat 2IAP from Scher Chemicals)
- 15 • 1,8-Bis (decyldimethylammonio)-3,6 dioxaoctane ditosylate

Nonionic Surfactants

- Ethoxylated glycerides
- monoglycerides such monoolein, monolinolein, monolaurin
- 20 Δ • diglyceride fatty acid (e.g., diglycerol monoisostearate Cosmol 41, fractionated, Nisshin Oil Mills Ltd.)
- Δ • Polyglyceryl esters (e.g., triglycerol monooleate (Grindsteal TS-T122), diglycerol monooleate (Grindsted TST-T101))
- Δ • Polyhydric alcohol esters and ethers (e.g., sucrose monooleate (Ryoto, Mitsubishi-Kasei Food Corp.) cetostearyl glucoside (Montanol, Seppic), β octyl glucofuranoside
- 25 esters, alkyl glucoside such C10-C16 (Henkel)
- Diesters of phosphoric acid (e. g., sodium dioleoyl phosphate)
- Ethoxylated alcohols (e.g., C16E8 (octaoxyethylene, glycol mono hexadecyl ether) and C12E8)
- 30 • Alkylamido propyl betaine (e.g., cocoamide propyl betaine)
- Amide: (e.g., N-(dodecanoylaminoethyl)-2-pyrrolidone)
- Amide oxide: e.g., 1,1 Dihydroperfluorooctyldimethylamine oxide
Dodecyldimethylamine oxide
2-Hydroxydodecyldimethylamine oxide
- 35 2-Hydroxydodecyl-bis (2-hydroxyethyl) amide oxide
2-Hydroxy-4-oxahexadecyldimethylamine oxide
- Ethoxylated amides (e.g., PEG-n acylamide)

Nonionic

- 40 • Ammonio phosphates (e.g., didecanoyl lecithin)
- Amine (e.g., octylamine)
- Ammonio amides e.g.,
N-trimethylammoniododecanamidate
N-trimethylammoniododecanamidate
- 45 • Ammonio carboxylates e.g.,
dodecyldimethylammonioacetate
6-didodecylmethylammoniohexanoate
- Monoglycerides e.g.,

- 1 dodecanoyl-glycerol monolaurin
- 1-13-docosenoyl-glycerol monoerucin
- Phosphonic and phosphoric esters and amides e.g.,
 - methyl-N-methyl-dodecylphosphonamidate
 - 5 dimethyl dodecylphosphonate
 - dodecyl methyl methylphosphonate
 - N,N-dimethyl dodecylphosphonic diamide
- Polyoxyethylene (C8) e.g.,
 - pentaoxyethylene Glycol p-n-octylphenyl ether
 - 10 hexaoxyethylene Glycol p-n-octylphenyl ether
 - nonaoxyethylene Glycol p-n-octylphenyl ether
- Polyoxyethylene (C10) e.g.,
 - pentaoxyethylene Glycol p-n-decylphenyl ether
 - decyl Glyceryl ether, 4-oxatetradecan-1,2-diol
 - 15 nonaoxyethylene glycol p-n-decylphenyl ether
- Polyoxyethylene (C11) e.g.,
 - Tetraoxyethylene glycol undecyl ether
- Polyoxyethylene (C12) e.g.,
 - 20 3,6,9,13-tetraoxapentacosan 1,11-diol
 - 3,6,10-trioxadocosan-1,8,diol
 - 3,6,9,12,16-pentaaoctacosan 1,14-diol
 - 3,6,9,12,15-pentaaxanonacosan-1,17-diol
 - 3,7-dioxanonadecan-1,5-diol
 - 25 3,6,9,12,15,19-hexaoxahentriacontan-1,16-diol
 - pentaoxyethylene glycol dodecyl ether
 - monaoxyethylene glycol p-n-dodecylphenyl ether
- Polyoxyethylene (C14) e.g.,
 - 3,6,9,12,16-pentaaoctacosan-1,14-diol
 - 30 3,6,9,12,115,19-heraoxatriacontan-1,17-diol
- Sulfone diimines e.g.,
 - decyl methyl sulfone diimine
- Sulfoxides e.g.,
 - 3-decyloxy-2-hydroxypropyl methyl sulfoxide
 - 35 4-decyloxy-3-hydroxybutyl methyl sulfoxide
- Sulfoximines e.g.,
 - N-methyl dodecyl methyl sulfoximine

Δ More preferred for use

- 40 Commercially available cationic surfactants suitable for use are: Abil B9950, Polysiloxane Betaine supplied by Goldschmidt; Arquat 2H-75 supplied by Akzo; Schercoquat 21AP supplied by Scher. Chem.; and Schercoquat DAS supplied by Scher Chem. Commercially available anionic surfactants suitable for use are: Crodafos N10 supplied by Croda and Dioctyl Sodium Sulfosuccinate supplied by American Cyanamid.
- 45 Commercially available nonionic surfactants suitable for use are: Crodesta F-50, Sucrose Distearate supplied by Croda; Diglycerol monoisostearate, Cosmol 41, Fractionated supplied by Nisshin; Dimodan DGMO and Triodan 20 supplied by

Grindsted; Generol 122 E-10 Ethoxylated Soya Sterol, Generol E-16 and Generol E-5 supplied by Henkel; Mirasoft MSP-011 supplied by Rhone-Poulenc; Montanol 68 Ecaïlles (cetearyl glucoside) supplied by Seppic; Sucrose Monooleate supplied by Mitsubishi; and Tetraglycerol Laurate supplied by Lonza. A commercially available
5 amphoteric surfactant suitable for use is Eldew CL-301 supplied by Ajinomoto.

The surfactants can be used at levels from about 4% to about 97%, preferably from about 5% to about 95%, more preferably from about 20% to about 90% and most preferably from about 30% to about 70% of the association structure.

Preparation of the Association Structure

10 Formation of the association structure, i.e., reverse micelles and/or liquid crystals and the concentration at which such association structures occur is dependent upon a variety of factors, including the specific types of surfactant, solvent, temperature, solubility of the surfactant in the solvent, and concentration of the surfactant in the carrier. The purity of the surfactant affects the concentration level at which the
15 association structures and particularly the preferred form of lamellar liquid crystals form.

The polar solvent and surfactant are mixed together. Formation of the association structure, particularly the preferred lamellar or hexagonal liquid crystalline state is accelerated by mechanical agitation. Mixing, can be performed either by hand (i.e., using hand utensils) or with mechanical equipment useful for home, institutional, or
20 industrial lipstick preparation. Extruders which provide a shearing operation with mixing can be used.

Generally the association structures are formed at ambient temperature/room temperature. The processing temperature will depend somewhat on the properties of the polar solvent. However, during processing the association structures will be
25 exposed to temperatures in the range of from about 10°C to about 100°C, preferably from about 70°C to about 90°C. If the temperatures affect the association structures, the association structures will reform once cooled to ambient temperature.

The one-phase liquid crystal is most preferred. It is preferred that a substantially two phase liquid crystal, one-phase liquid crystal or single phase liquid crystal
30 component of (preferably at least 90%) be utilized.

Separation and thus detection of the association structure from excess liquid (solvent or solution) or solid may be achieved by ultracentrifugation. Ultracentrifugation should be conducted using sufficiently high centrifugal forces (preferably within the range of from about 20,000 rpm to about 60,000 rpm for from
35 about one hour to about sixteen hours utilizing a Beckman L8-80 centrifuge equipped with a SW60Ti Rotor or by applying about 300,000*g for about one hour) to induce the formation of observable phase boundaries over a period of time. Under these conditions a good separation of the individual phases is obtained. The volume of each phase is determined by calibration of the centrifuge tube and the volume fraction of the individual

phase thus calculated.

Wax

The wax acts as a solidifying agent in the lipstick. It assists in the formation of the solid structure of the lipstick. The wax is comprised of organic compounds or mixtures of high molecular weight substances, and is solid at ambient temperature/room temperature. The wax can be hydrocarbons or esters of fatty acids and fatty alcohols. Waxes are thermoplastic. Natural, mineral and synthetic waxes can be used herein. As used herein "wax" refers to mixtures as well as a single type of wax.

Natural waxes can be of animal origin, e.g. beeswax, spermaceti, lanolin, shellac wax, or of vegetable origin, e.g. carnauba, candelilla, bay berry, sugarcane wax, and of mineral origin, e.g. ozokerite, ceresin, montan, paraffin, microcrystalline wax, petroleum and petrolatum wax. Synthetic waxes include polyol ether-esters such as carbowax and hydrocarbon-type waxes, silicone waxes and polyethylene wax. Generally, the waxes useful herein have melting points from about 55°C to about 110°C and are selected from the C₈ to C₅₀ hydrocarbon waxes.

The preferred waxes of the present invention are selected from the group consisting of candelilla, beeswax, carnauba, spermaceti, montan, ozokerite, ceresin, paraffin, modified beeswax, bayberry, castor waxes, synthetic waxes, microcrystalline waxes and mixtures thereof. More preferably the waxes are selected from the group consisting of microcrystalline, spermaceti, candelilla, modified beeswax, carnauba, ozokerite, paraffin, ceresin, and mixtures thereof. Most preferably, the waxes are selected from the group consisting of microcrystalline, candelilla, modified beeswax, ozokerite, paraffin, and mixtures thereof. A particularly preferred mixture of waxes used in the present invention is as follows:

- a. from about 3% to about 6% candelilla wax;
- b. from about 2% to about 5% ozokerite wax;
- c. from about 2% to about 5% paraffin wax; and
- d. from about 1% to about 4% microcrystalline wax.

The amount of wax used is from about 5% to about 90%, preferably from about 10% to about 30% and most preferably from about 10% to about 20% of the lipstick composition.

Emollient Component

The emollient component can comprise fats, oils, fatty alcohols, fatty acids and esters which aid application and adhesion, yield gloss and most importantly provide occlusive moisturization. The removal of castor oil from the preferred lipstick compositions of the present invention allows the utilization of more emollients.

Suitable emollients for use are isostearic acid derivatives, isopropyl palmitate, surfactants, lanolin oil, diisopropyl dimerate, maleated soybean oil, octyl palmitate, isopropyl isostearate, octyl hydroxystearate, cetyl lactate, cetyl ricinoleate, tocopheryl

acetate, acetylated lanolin alcohol, cetyl acetate, lecithin, phenyl trimethicone, glyceryl oleate, tocopheryl linoleate, wheat germ glycerides, arachidyl propionate, isopropyl palmitate, myristyl lactate, decyl oleate, propylene glycol ricinoleate, isopropyl lanolate, pentaerythrityl tetrastearate, neopentylglycol dicaprylate/dicaprate, hydrogenated coco-
5 glycerides, isotridecyl isononanoate, myristal myristate, triisocetyl citrate, cetyl alcohol, octyl dodecanol, oleyl alcohol, panthenol, lanolin alcohol, linoleic acid, linolenic acid and mixtures thereof.

Polar solvent/moisturizing agents in excess of the polar solvents forming association structures can be used in the emollient component. When these excess polar
10 solvents are utilized in the emollient component, it is preferred that a coupling agent also be used. Glycerine is a preferred moisturizer and preferably forms a part of the association structure system. Other preferred moisturizers include pyrrolidone carboxylic acid, sodium lactate or lactic acid, urea collagen, α -hydroxy propylglyceryl ether α -hydroxy acids (e.g., ethylglycolic acid, leucic acid, mandelic acid, glycollic acid),
15 glucosamines, and elastin fibers, D-panthenol, aklantoin and hyaluronic acid and chondroitin sulfate. Please note that some of these can be delivered with the association structures by dissolving into the polar liquid.

Oils are those materials which are organic substances that are liquid at ambient temperature. They are esters, triglycerides, hydrocarbons and silicones. These can be a
20 single material or a mixture of one or more materials. They will normally comprise from 0% to about 100%, preferably from about 5% to about 90%, and most preferably from about 70% to about 90% of the emollient component.

Preferably, the oils are minimized in the present invention due to their tendency to sweat. A preferred embodiment of the present invention utilizes a coupling agent when
25 the compositions comprise greater than about 40% oil. The oils act as emollients and also impart viscosity, tackiness, and drag properties to the lipstick. Suitable oils include caprylic triglycerides; capric triglyceride; isostearic triglyceride; adipic triglyceride; propylene glycol myristyl acetate; lanolin; lanolin oil; polybutene; isopropyl palmitate; isopropyl myristate; isopropyl isostearate; diethyl sebacate; diisopropyl adipate;
30 tocopheryl acetate; tocopheryl linoleate; hexadecyl stearate; ethyl lactate; cetyl oleate; cetyl ricinoleate; oleyl alcohol; hexadecyl alcohol; octyl hydroxystearate; octyl dodecanol; wheat germ oil; hydrogenated vegetable oils; petrolatum; modified lanolins; branched-chain hydrocarbons; alcohols and esters; corn oil; cottonseed oil; olive oil; palm kernel oil; rapeseed oil; safflower oil; jojoba oil; evening primrose oil; avocado oil
35 mineral oil, sheabutter, octylpalmitate, maleated soybean oil, glycerol trioctanoate, diisopropyl dimerate, and volatile and non-volatile silicone oils including phenyl trimethicone. Castor oil is not preferred for use herein. Preferred lipstick composition of the present invention are substantially free of castor oil such that the lipstick comprises less than about 0.1%, preferably less than about 0.01% and most preferably

less than about .001% of castor oil.

The preferred oils for use herein are caprylic triglycerides, capric triglycerides, isostearic triglyceride, adipic triglyceride, phenyl trimethicone, lanolin oil, polybutene, isopropyl palmitate, isopropyl isostearate, cetyl ricinoleate, octyl dodecanol, oleyl
5 alcohol, hydrogenated vegetable oils, modified lanolins octyl palmitate, lanolin oil, maleated sooybean oil, cetylricinoleate, glyceryltriocanoate diisopropyl dimerate, synthetic anolin derivatives and branched chain alcohols and mixtures thereof.

Preferably, the oils used are selected such that the majority (at least about 75%, preferably at least about 80% and most preferably at least about 99%) of the types of
10 oils used have solubility parameters which do not differ by more than from about 1 to about .3, preferably from about .8 to about .5. For example, the more preferred oils for use are lanolin oil, octyl palmitate and isopropylpalmitate. Their respective solubility parameters are 7.3, 7.4 and 7.8. Thus, the solubility parameters do not differ by more than about .5. (Solubility parameters as reported in "Cosmetics & Toletries", Vol 103,
15 October 1988, p64.) It is also preferred that the oils and waxes utilized have compatible solubilities.

The more preferred oils for use herein have a solubility parameter of from about 7.3 to abut 7.8. Examples of more preferred oils for use herein are lanolin oil, octyl palmitate, isopropylpalmitate and mixtures threof.

20 The emollient component comprises from about 1% to about 90%, preferably from about 10% to about 80%, more preferably from about 20% to about 70%, and most preferably from about 40% to about 60%, of the lipstick composition.

Color

The lipsticks can contain from 0% to about 35% preferably from about 1% to
25 about 20% and most preferably from about 5% to about 15%, of color, on an anhydrous pigment weight basis. These are usually aluminum, barium or calcium salts or lakes. Preferably, dyes are present at from about 0.1% to about 4% and pearls from 0% to about 20%. Colors which are dispersed in castor oil are not preferred for use.

Preferably, the lipstick compositions of the present invention are substantially free
30 of castor oil such that the lipstick comprises less than about 0.1%, preferably less than about 0.01% and most preferably less than about .001% castor oil.

Pigments are typically dispersed in castor oil for the good dispersion of the pigments when incorporated into the lipstick, thus providing an even distribution of color. It has been discovered that excellent dispersion of the pigment can be achieved by
35 utilizing the association structures, preferably lamellar liquid crystals, as a means of incorporating the color/pigments into the lipstick. A preferred method of incorporating dry pigments comprises the steps of:

(a) preparing a mixture consisting essentially of:

(1) a polar solvent; and

- (2) a surfactant selected from the group consisting of amphoteric, cationic, anionic and nonionic surfactants having a Krafft point at or below about ambient temperature and mixtures thereof; and
- (b) stirring said mixture until association structures form;
- 5 (c) adding and mixing dry pigments until achieving a homogenous mixture;
- (d) milling said mixture until uniform particle size is achieved; and
- (e) adding and mixing the mixture of (c) to the remaining lipstick ingredients until a homogenous mixture is obtained.

If the ingredients of the lipstick composition are being processed such that the association structures are being formed in situ, the preferred method of incorporating the dry pigments is to slurry them in one or more of the liquid emollient ingredients.

It should be noted that during processing of the association structure lipstick compositions, there is an improvement in the form of a noted decrease in the amount of separation of pigment particles during processing and molding.

15 Colors/pigments suitable for use herein are all inorganic and organic colors/pigments suitable for use in lipstick compositions.

Lakes are either a pigment that is extended or reduced with a solid diluent or an organic pigment that is prepared by the precipitation of a water-soluble dye on an adsorptive surface, which usually is aluminum hydrate. There is uncertainty in some instances as to whether the soluble dye precipitates on the surface of the aluminum hydrate to yield a dyed inorganic pigment or whether it merely precipitates in the presence of the substrate. A lake also forms from precipitation of an insoluble salt from an acid or basic dye. Calcium and barium lakes are also used herein.

Preferred lakes of the present invention are Red 3 Aluminum Lake, Red 21 Aluminum Lake, Red 27 Aluminum Lake, Red 28 Aluminum Lake, Red 33 Aluminum Lake, Yellow 5 Aluminum Lake, Yellow 6 Aluminum Lake, Yellow 10 Aluminum Lake, Orange 5 Aluminum Lake and Blue 1 Aluminum Lake, Red 6 Barium Lake, Red 7 Calcium Lake.

Other colors and pigments can also be included in the lipsticks, such as dyes and pearls, titanium oxides, Red 6, Red 21, Brown, Russet and Sienna dyes, chalk, talc, iron oxides and titanated micas.

Other Additives

Optional ingredients which can be present in the lipstick include the flavor oils which were described above, fat soluble vitamins such as vitamin A and E, sunscreens and skin care actives. The lipstick can comprise ingredients conventionally employed in lipsticks or other lipcare products. This includes skin care active ingredients such as pharmaceutically active ingredients.

Flavor oils such as peppermint oil, orange oil, citrus oil, wintergreen oil can be used along with an alcohol or glycerine. Flavor oils are usually mixed in a solvent such

as ethanol to dilute the flavor. The flavor oils useful herein can be derived from natural sources or be synthetically prepared. Generally flavor oils are mixtures of ketones, alcohols, fatty acids, esters and terpenes. The term "flavor oil" is generally recognized in the art to be a liquid which is derived from botanical sources, i.e. leaves, bark, or skin of fruits or vegetables, and which are usually insoluble in water. The level of flavor oil used can range from 0% to about 5%, preferable from 0% to about 1%.

Emulsifiers do not form association structures at ambient temperature with the polar solvent utilized therein can also be used. The overall concentration of the emulsifier can be from 0% to about 20% of the formulation, preferably from 0% to about 15% and most preferably from about 1% to about 10%.

These emulsifiers are used as a coupling agents which have an affinity for the hydrophilic (not the polar solvent) and hydrophobic phases of the lipsticks, yet do not form association structures at ambient temperature. Examples of suitable coupling agents are sorbitan oleate, sorbitan sesquioleate, PG-3 diisostearate, dipentaerythritol fatty acid ester, cholesterol 12 hydroxystearate, and mixtures thereof.

A preferred embodiment of the present invention comprises from about 0.1% to about 30%, preferably from about 8% to about 15%, polar solvent and from about 5% to about 20%, surfactants of the lipstick composition. The surfactants are preferably a mixture wherein from about 50% to about 75% of the mixture is made up of surfactants which have a Krafft point of at or below about ambient temperature and form association structures at ambient temperature and from about 25% to about 50% of the mixture is made up of surfactants which are coupling agents. Another preferred mixture of surfactants which can form association structures and surfactants which act as coupling agent is lecithin, PG-3 diisostearate, sorbitonleate, cholesterol 12 hydroxystearate and dipentaerythritol fatty acid ester. Another preferred mixture is dipentaerythritol fatty acid ester, lecithin, and PG-3 diisostearate.

Skin care actives ingredients in both water soluble and water insoluble forms can be added to the lipstick. These include zinc oxide, beta-glycyerhetic acid; chamomile oil; ginko biloba extract; pyroglutamic acid, salts or esters; sodium hyaluronate; 2-hydroxyoctanoic acid; sulfur; salicylic acid; carboxymethyl cysteine, and mixtures thereof. These will normally be present in amounts of less than about 2% by weight, and generally in the range of about .01% to about 1% by weight.

A preferred optional component is ethyl cellulose (Ethocel). Ethyl cellulose generally is preferred for use at levels of about 5% and more preferably 1%.

Another preferred optional component is silica. Silica is generally preferred for use at levels of from about 1% and about 5%.

Hypoallergenic Lipsticks

Hypoallergenic lipsticks can be made from the liquid crystal, wax, oil and colors herein. These lipsticks should not contain fragrances, flavor oils, lanolin, sunscreens,

particularly PABA, or other sensitizers and irritants.

Addition of the association structure to lipsticks

The association structure can be used in conventional lipstick formulating as a substitute for castor oil, other oils, and other lipstick ingredients. The association
5 structures can be formed before addition of the polar solvent component and surfactant component of the association structure can be added independently and the association structures will form in situ. Preferably from 10% to 60%, preferably from about 20% to about 50%, of the oil or wax component is replaced with the stable liquid crystal. Generally lipstick formulations can be adjusted without undue experimentation.

10 The association structure should be well mixed with the solid component of the composition. It is preferable to prepare the association structures first, preferably liquid crystals or reverse hexagonal liquid crystals and more preferably lamellar liquid crystals, and then mix the association structures with the waxes and oils in order to most effectively achieve a microscopic distribution of the association structure in the solid.

15 The association structures, preferably lamellar liquid crystals and/or reverse hexagonal liquid crystals, can be mixed with the waxes while they are molten and the mixture molded by conventional means. Preferably, the waxes and emollient component are melted at a temperature of from about 70°C to about 95°C, preferably from about 83°C to 90°C and the association structure is added with stirring. The mixture is then
20 poured into a mold at room temperature. The molding temperature can be varied to give a more uniform stick. Other conventional lipstick making processes can be used.

The following examples illustrate the invention but are not intended to be limiting thereof.

Example I

25 A lipstick composition of the present invention which is substantially free of castor oil, is prepared as follows:

	<u>Ingredient</u>	<u>Amount (weight percent)</u>
	Carnauba	1.50
30	Ozokerite	6.00
	Candelilla	4.00
	Hydrogenated Vegetable Oil	5.00
	Acetylated Lanolin	4.00
	Isopropyl Isostearate	11.90
35	Isostearic Acid	10.00
	Propylparaben	0.10
	Cetyl Ricinoleate	10.00
	Ascorbyl Palmitate	1.00
	Silica L-700	1.00
40	Polybutene	2.00
	Petrolatum	5.50

21

Association Structure Phase

	Sucrose Monooleate	12.00
	Panthenol	5.00
	Glycerine	12.00
5	Pigment	9.00
	Total	100.00

The ingredients for the Association Structure Phase, except for the pigments, are mixed until association structures are formed. Once the association structures are formed, the pigments are added and milled on a three roll mill. The mixture is then
 10 mixed with the other ingredients to make a homogeneous mixture. This mixture is heated to 85°C and then poured into a mold at room temperature.

Example II

A lipstick composition of the present invention which is substantially free of castor oil, is prepared as follows:

15	<u>Ingredient</u>	<u>Amount (weight percent)</u>
	Carnauba	1.50
	Ozokerite	6.00
	Candelillia	4.00
	Hydrogenated Vegetable Oil	5.00
20	Isopropyl Palmitate	11.90
	Isostearic Acid	10.00
	Acetylated Lanolin	4.00
	Propylparaben	0.10
	Cetyl Ricinoleate	10.00
25	Ascorbyl Palmitate	1.00
	Silica L-700	1.00
	Polybutene	2.00
	Petrolatum	5.50
	<u>Association Structure Phase</u>	
30	Sucrose Monooleate	12.00
	Panthenol	5.00
	Glycerine	12.00
	Pigment	9.00
	Total	100.00

35 The composition is prepared as in Example I.

Example III

A lipstick composition of the present invention which is substantially free of castor oil, is prepared as follows:

	<u>Ingredient</u>	<u>Amount (weight percent)</u>
40	Carnauba	1.50
	Ozokerite	5.50
	Candelillia	4.00
	Hydrogenated Vegetable Oil	5.00
	Acetylated Lanolin	4.00
45	Propylparaben	0.10
	Cetyl Ricinoleate	10.00

		22
	Ascorbyl Palmitate	1.00
	Polybutene	2.00
	Polysiloxane Copolymer ¹	5.97
	Petrolatum	5.97
5	Anhydrous Lanolin	5.97

Association Structure Phase

	Lecithin	22.95
	Panthenol	5.04
10	Glycerine	12.00
	Pigment	9.00
	Total	100.00

¹ #1154-141-1, supplied by GE Silicones.

The composition is prepared as in Example I.

15 Example IV

A lipstick composition of the present invention is prepared as follows:

	<u>Ingredient</u>	<u>Amount (weight percent)</u>
	<u>Waxes:</u>	
	Ozokerite	3.50
20	Paraffin	3.25
	Candelilla Wax	4.65
	Microcrystalline Wax	3.00
	<u>Oils:</u>	
	Octyl Palmitate	13.50
25	Lanolin Oil	8.50
	Isopropyl Palmitate	8.50
	Maleated Soybean Oil	2.00
	Cetyl Ricinoleate	4.00
	<u>Pigment Phase:</u>	
30	Diisopropyl Dimerate	12.00
	Pigment	12.00
	<u>Surfactants/Emulsifiers:</u>	
	Lecithin (CentrolexF)	0.70
	PG-3 Diisostearate	3.25
35	Sorbitan Oleate	5.00
	Cholesterol 12 Hydroxystearate	2.00
	Dipentaerythritol Fatty Acid Ester	4.00
	<u>Polar Solvents:</u>	
	Glycerine	9.00
40	Panthenol	1.00
	<u>Miscellaneous:</u>	
	Tocopherol	0.10
	Propylparaben	0.05
45	Total	100.00

The pigment is slurried in the diisopropyl dimerate. The waxes and oils are placed in a beaker and melted in a steam bath at about 85°C. When the wax solids have almost melted, the mixture is mixed for twenty to thirty minutes. After mixing the beaker is

- removed from the steam bath and the mixture/base checked for clarity and uniformity. If some particles remain, the base should be mixed again until the particles/solids are no longer visible. The mixture is allowed to solidify. The other ingredients including the pigment slurry are mixed with a straight stainless steel spatula until uniform. The mixture is added to the base and melted over a steam bath and mixed for fifteen to twenty minutes. Once uniform, the beaker is removed and the composition poured into molds at room temperature.

Example V:

A lipstick composition of the present invention is prepared as follows:

10	<u>Ingredient</u>	<u>Amount (weight percent)</u>
	<u>Phases A:</u>	
	Ozokerite	4.45
	Candelilla Wax	5.25
	Be Square-175	1.65
15	Paraffin	3.25
	Phenyl Trimethicone	4.50
	Octyl Palmitate	10.00
	Isopropyl Palmitate	8.00
	Glyceryl Trioctanoate	12.00
20	Maleated Soybean Oil	2.00
	Diisopropyl Dimerate	8.00
	<u>Phase B:</u>	
	Dipentaerythritol Fatty Acid Ester ¹	4.50
	PG-3 diisostearate	3.00
25	Lecithin	0.50
	Tocopherol Acetate	0.05
	Propylparaben	0.10
	Glycerine	9.00
	Panthenol	1.00
30	<u>Phase C:</u>	
	Pigment	11.37
	Diisopropyl dimerate	11.38
	Total	100.00

- 35 ¹ Cosmol 168Ar supplied by Nisshin Oil Mills, LTD.

The ingredients of Phase A are mixed together over a steam bath at about 85°C to melt the solids. The ingredients of Phase B are mixed together and then added to Phase A which has been allowed to resolidify once a homogeneous mixture was obtained

(about 20 to 30 minutes of mixing over steam bath). The mixture of Phase A and B is melted over a steam bath and mixed for about 15 to 20 minutes. The ingredients of Phase C are slurried and added to the mixture of A & B. Mix until a homogenous mixture is obtained then remove from the steam bath and pour into molds.

5

Example VI

A lipstick composition of the present invention, which is substantially free of castor oil, is prepared as follows:

	<u>Ingredient</u>	<u>Amount</u> (weight percent)
	Carnauba	1.50
10	Ozokerite	5.50
	Candellia	4.00
	Hydrogenated Vegetable Oil	5.00
	Oleyl Alcohol	5.00
	Isopropyl Isostearate	7.90
15	Acetylated Lanolin	4.00
	Propylparaben	0.10
	Cetyl Ricinoleate	6.00
	Ascorbyl Palmitate	1.00
	Polybutene	2.00
20	<u>Association Structure Phase</u>	
	Lecithin	33.00
	Panthenol	5.00
	Glycerine	12.00
	Pigment	<u>9.00</u>
25		100.00

The composition is prepared as in Example I.

Example VII

A lipstick composition of the present invention is prepared as follows:

	<u>Ingredient</u>	<u>Amount</u> (weight percent)
30	Carnauba	1.50
	Ozokerite	5.50
	Candelilla	4.00
	Hydrogenated Vegetable Oil	3.00
35	Acetylated Lanolin	6.00
	Isopropyl Isostearate	12.00
	Propylparaben	0.10
	Oleyl Alcohol	3.90
	Cetyl Ricinoleate	7.00
40	Ascorbyl Palmitate	1.00
	Polybatene	2.00

25

Association Structure Phase

	Cetostearyl glucoside ¹	7.20
	Glycerine	12.00
	Panthenol	4.80
5	Color slurry (pigment and castor oil)	<u>30.00</u>
		100.00

¹ Cetostearyl glucoside is a nonionic surfactant supplied as Montanol 68 Ecailles supplied by Seppic.

- 10 The waxes and oils are mixed together over a steam bath at about 85°C until a homogeneous mixture is achieved. This base mixture is allowed to solidify. The remaining ingredients are mixed together then added to the base and placed over a steam bath (about 85°C). The resulting mixture is mixed over the steam bath until a homogeneous mixture is achieved and then poured into molds and cooled.

15 Example VIII

A lipstick composition of the present invention is prepared as follows:

	<u>Ingredient</u>	<u>Amount (weight percent)</u>
	Carnauba	1.50
	Ozokerite	5.50
20	Candellia	4.00
	Hydrogenated Vegetable Oil	5.00
	Acetylated Lanolin	6.00
	Isopropyl Isostearate	10.50
	Propylparaben	0.10
25	Oleyl alcohol	5.40
	Cetyl Ricinoleate	5.00
	Ascorbyl Palmitate	1.00
	Polybutene	2.00
	<u>Association Structure Phase</u>	
30	Schercoquat 21AP ¹	12.00
	Glycerine	12.00
	Color slurry:	
	Pigment and Castor oil	<u>30.00</u>
		100.00

35

¹ A cationic surfactant, Bis Isostearamidopropyl Hydroxypropyl diammonium chloride supplied by Scher Chemicals Inc.

The composition is prepared as in Example VII.

Example IX

- 40 A lipstick composition of the present invention comprising liquid crystal association structures is prepared as follows:

	<u>Ingredient</u>	<u>Amount (weight percent)</u>
	Carnauba	1.50
	Ozokerite	5.50

26

	Candelilla	4.00
	Hydrogenated Vegetable Oil	5.00
	Acetylated Lanolin	4.00
	Isopropyl Isostearate	10.50
5	Propylparaben	0.10
	Oleyl Alcohol	3.40
	Cetyl Ricinoleate	5.00
	Ascorbyl Palmitate	1.00
	Polybutene	2.00
10	<u>Liquid Crystal Phase</u>	
	Centrox F*	11.20
	Glycerine	12.00
	Panthenol	4.8
	Color Slurry	30.00
15	Total	100.00

*(from Central Soya)

The Centrox F, glycerine and panthenol are mixed until a liquid crystal phase (L.C.) is formed. The color slurry is a mixture of pigments (30%) slurried in castor oil (70%). The neat single phase liquid crystal is then mixed with the other ingredients to make a homogeneous mixture. This mixture is heated to 80°C and then poured into a mold at room temperature.

Example X

A lipstick composition of the present invention comprising liquid crystal association structures is prepared as follows:

25	<u>Ingredient</u>	<u>Amount (weight in grams)</u>
	<u>Base</u>	
	Carnauba	11.25
	Ozokerite	18.00
	Hydrogenated Vegetable Oil	18.00
30	Acetylated Lanolin	15.75
	Isopropyl Isostearate	45.00
	Propylparaben	0.23
	Oleyl Alcohol	20.25
	<u>Liquid Crystal Phase</u>	
35	Centrox F	18.00
	Glycerine	27.00
	<u>Color</u>	

27

Castor Oil	31.28
Pigments	20.25
Total	225.00

Centrox F is an essentially oil-free lecithin powder from Central Soya (Fort Wayne, IN).

5 The lipstick is prepared as in Example IX_{to}

Example XI

A lipstick composition of the present invention comprising liquid crystal association structures is prepared as follows:

<u>Ingredient</u>	<u>Amount (weight percent)</u>
10 <u>Base</u>	
Carnauba	3.60
Ozokerite	5.76
Hydrogenated Vegetable Oil	5.76
Acetylated Lanolin	5.04
15 Isopropyl Isostearate	9.04
Propylparaben	0.07
Oleyl Alcohol	2.37
Cetyl Ricinoleate	10.00
<u>Liquid Crystal Phase</u>	
20 Centrox F (Lecithin)	11.20
Glycerine	12.04
Panthenol	4.76
Castor Oil	21.00
Pigments	9.00

25 The panthenol is dissolved in heated glycerine and mixed with the Centrox F to form a single phase liquid crystal. The liquid crystal is then added to remaining ingredients as in Example IX. A glossy and slippery lipstick is formed.

Example XII

30 A lipstick composition of the present invention comprising liquid crystal association structures is prepared as follows:

<u>Ingredient</u>	<u>Amount (weight percent)</u>
<u>Waxes and Oils</u>	
Carnauba	3.60
Ozokerite	5.76
35 Hydrogenated Vegetable Oil	5.76
Acetylated Lanolin	5.04
Isopropyl Isostearate	9.40
Propylparaben	0.07
Oleyl Alcohol	2.37

28

	Silica	5.00
	Cetyl Ricinoleate	5.00
	<u>Liquid Crystal Phase</u>	
	Centrolux F	40.00
5	Glycerine	42.85
	Panthenol	17.15
	Castor Oil	21.00
	Lake	9.00
	Total	100.00

10

The lipstick is prepared as in Example XI.

Identification of Association Structures

Those skilled in the area of association structures will be able to identify
15 association structures based upon known identification techniques.

In identifying association structures, it is preferred that the individual selected
surfactants be combined with glycerine or water over a concentration range at about
ambient temperature to determine if the individual selected surfactants are capable of
forming association structures. When combined, surfactants and polar solvents will not
20 form in the product if the selected surfactants do not form association structures at some
concentration with glycerine or water at about ambient temperature. Well known
identification techniques can be used on the mixture of the individual selected surfactants
and glycerine or water.

Association structure formation for any particular surfactant and solvent
25 combination is readily identified using one or more of several well known identification
techniques. The onset of association structure formation and in particular the
occurrence of the most preferred substantially one-phase liquid crystal state for a
particular phosphatide or surfactant and solvent system can be identified by: (1) visual
observation with the naked eye, (2) birefringent optical activity observed by light
30 microscopy; (3) measurement of the phosphatide or surfactant/solvent system NMR
spectra; (4) x-ray diffraction; (5) presence of a characteristic "texture" pattern
observable under polarized light microscopy; and/or (6) texture observed in freeze
fractured micrographs by transmission electron microscopy (TEM). Typically, polarized
light microscopy determination requires confirmation by one of the other above
35 mentioned methods. Light microscopy of liquid crystals is described generally in The
Microscopy of Liquid Crystals, Norman, Hartshorn, London, England and Chicago,
Illinois, U.S.A., 1974, which discusses birefringence of mesomorphic states and methods
for microscopic observation and evaluation (Chapter 1, pp. 1-20). Birefringence is a
preferred method for determining the occurrence of a liquid crystal.

The identification of association structures within the lipstick product is generally more difficult due to the presence of other compounds such as wax crystals or pigments. Thus, the preferred way for identification of association structures such as liquid crystals is to ultracentrifuge the lipstick sample as previously described, separate the layers, identify the layer with typical association structure birefringence and submit that layer to testing by x-ray diffraction and/or transmission electron microscopy (TEM). Freeze-fracture transmission electron microscopy (FF/TEM) is the more preferred method of identification. Most preferably, FF/TEM is utilized to confirm association structures which have been indicated by other well-known methods such as x-ray diffraction or NMR.

A preferred method for determining the occurrence of the association structures of the present invention is by transmission electron microscopy (TEM). More preferably, the association structures are imaged by a freeze-fracture transmission electron microscopy (FF/TEM) method. The method is carried out as follows:

1. The outside cavity of a freezing container is filled with liquid nitrogen and the inner dewar of the freezing container is filled with liquid ethane (normal melting temperature of -172°C). The ethane is allowed to freeze.
2. A small piece (1mm X 2mm) is cut from the lipstick with a clean razor blade and placed in the well of a copper specimen holder.
3. Most of the frozen ethane in the dewar is melted by inserting a metal heat sink into the dewar.
4. Immediately after melting the ethane, the specimen holder containing the lipstick sample is picked up using a pair of tweezers and rapidly plunged into the liquid ethane.
5. After a few seconds, the specimen holder is removed from the ethane, quickly touched to the tip of a camel's hair brush to remove excess ethane, and immediately immersed in the liquid nitrogen to keep the sample cold.
6. The sample is transferred under liquid nitrogen to a JEOL JFD-9000C sample holder and then transferred into the chamber of a JEOL JFD-9000C freeze fracture unit. The temperature of the specimen stage in the unit should be about -175°C . Vacuum should be at least 5×10^{-7} torr.
7. A knife inside the unit is cooled to a temperature of about -165°C .

8. The sample is fractured in the JEOL chamber using the pre-cooled knife.
9. Platinum-carbon is deposited onto the fractured sample at a 45° angle for 4.5
5 seconds, followed by carbon deposition at a 90° angle for 25 seconds to form a replica
of the fractured sample. The high voltage is 2500V and the current is 70mA.
10. The samples are removed from the freeze-fracture unit and cleaned in
subsequent solutions of warm Dawn® (a liquid dishwashing detergent sold by The
10 Procter and Gamble Company) in water, methanol, chloroform/methanol, and
chloroform to remove the sample from the replica.
11. The replicas are picked up on 300 mesh copper EM grids and examined in a
transmission electron microscope.
- 15 12. Images are recorded on negative film and positive prints are made from the
negatives.
13. The prints are then examined by one of ordinary skill in the art for identification
20 based upon known identification techniques.

The freeze-fracture transmission electron microscopy method is described generally in the following references which are incorporated herein by reference: Rash, J.E. and Hudson, C.S., Freeze-Fracture: Methods, Artifacts, and Interpretations,
25 New Haven Press, New York, 1979; and Steinbrecht and Zierold, Cryotechniques in Biological Electron Microscopy, Springer-Verlag, Berlin, 1987. The use of the freeze-fracture transmission electron microscopy method for structure determination and identification is generally described in the following references which are incorporated herein by reference: Gulik-Krzywicki, T., Aggerbeck, L.P. and Larsson, K., "The use
30 of Freeze-Fracture and Freeze-Etching Electron Microscopy for Phase Analysis and Structure Determination of Lipid Systems," Surfactants in Solution, K.L. Mittal and B. Lindman, eds., Plenum Press, New York, pp. 237-257, 1984; and Zasadzinski, J.A.N. and Bailey, S.M., "Applications of Freeze-Fracture Replication to Problems in Materials and Colloid Science," J. Elect. Micros. Tech., 13:309-334, 1989

5 CLAIMS

1. A lipstick composition comprising:
- (a) from 5% to 90%, preferably 10% to 30%, by weight, wax;
 - (b) from 1% to 90%, preferably 10% to 80%, by weight, of an emollient component comprising from 0% to 100%, preferably 5% to 90%, by weight, of oil which is liquid at ambient temperature;
 - (c) from 0.1% to 80%, preferably from 3% to 75%, and more preferably from 10% to 65%, by weight, of an association structure consisting essentially of:
 - (1) from 3% to 96%, preferably from 10% to 80%, by weight, of polar solvent preferably selected from the group consisting of water, glycerine, propylene glycol, butylene glycol, panthenol and mixtures thereof; and
 - (2) from 4% to 97%, preferably from about 30% to about 80%, by weight, of surfactant having a Krafft point at or below ambient temperature preferably selected from the group consisting of amphoteric surfactants, anionic surfactants, cationic surfactants, nonionic surfactants and mixtures thereof; and
 - (d) from 0% to 35%, preferably 1% to 20%, on an anhydrous basis, color.
2. A lipstick composition according to claim 2 wherein said association structure is selected from the group consisting of reverse micelles, lyotropic liquid crystals and mixtures thereof; preferably from the group consisting of cylindrical reverse micelles, reverse hexagonal liquid crystals, cubic liquid crystals, lamellar liquid crystals and mixtures thereof; and more preferably from the group consisting of lamellar liquid crystals, reverse hexagonal liquid crystals and mixtures thereof.
3. A lipstick composition according to claim 2 wherein said reverse micelles aggregate to form networking spherical structures, elongated structures cylindrical structures, filament structures or mixtures thereof.
4. A lipstick composition according to claim 2 wherein said lamellar liquid crystals are substantially one phase.

- 5 5. A lipstick composition according to claim 2 wherein said lipstick composition is substantially free of castor oil.
6. A lipstick composition according to claim 2 wherein said lipstick composition is substantially free of water.
- 10 7. A lipstick composition according to claim 2 wherein said surfactant is selected from the group consisting of amphoteric surfactants, cationic surfactants, nonionic surfactants and mixtures thereof.
- 15 8. A lipstick composition according to claim 7 wherein said surfactant is selected from the group consisting of nonionic surfactants.
9. A lipstick composition according to claim 8 wherein said oil is selected such that at least 75%, preferably 99%, of the types of oils used have solubility parameters which do not differ by more than from .3 to 1, preferably from 20 0.5 to .8.
10. A lipstick composition according to claim 2 wherein said wax is selected from the group consisting of:
- 25 candelilla,
beeswax,
carnauba,
spermaceti,
montun,
30 ozokerite,
ceresin,
paraffin,
modified beeswax,
bayberry,
35 castor waxes,
synthetic waxes,
microcrystalline waxes, and mixtures thereof and preferably said wax is selected from the group consisting of microcrystalline waxes, candelilla, modified beeswax, ozokerite, paraffin and mixtures thereof..
- 40

- 5 11. A lipstick composition according to claim 10 wherein said lipstick composition comprises from 3% to 6% candelilla wax, from 2% to 5% ozokerite wax, from 2% to 5% paraffin wax, and from 1% to 4% microcrystalline wax.
- 10 12. A lipstick composition according to claim 11 wherein said lipstick composition is substantially free of lecithin.
13. A lipstick composition according to claim 2 further comprising from 1% to 10% of a coupling agent.
- 15 14. A lipstick composition comprising:
- (a) from 5% to 90%, preferably 10% to 30%, by weight, wax;
 - (b) from 1% to 90%, preferably 10% to 80%, by weight, of an emollient component comprising from 0% to 100%, preferably 5% to 90%, by weight, of oil which is liquid at ambient temperature;
 - 20 (c) from 0.1% to 80%, preferably from 3% to 75%, and more preferably from 10% to 65%, by weight, of an association structure consisting essentially of:
 - 25 (1) from 0.1% to 30%, by weight, of polar solvent preferably selected from the group consisting of water, glycerine, propylene glycol, butylene glycol, panthenol and mixtures thereof; and
 - (2) from 5% to 20%, by weight, of surfactant wherein said surfactant is a mixture having from 50% to 75% of the mixture being surfactants which have a Krafft point at or below ambient temperature and form association structures at ambient temperature and from about 25% to about 50% of the mixture being surfactants which are coupling agents;
 - 30 (d) from 0% to 35%, preferably 1% to 20%, on an anhydrous basis, color.
- 35 15. A process for incorporating dry pigments into a lipstick composition comprising the steps of:
- (a) preparing a mixture consisting essentially of:
 - (1) a polar solvent; and
 - (2) a surfactant selected from the group consisting of amphoteric surfactants, cationic surfactants, anionic surfactants, nonionic surfactants having a Krafft point at or below about ambient temperature and mixtures thereof;
 - 40

- 5 (b) stirring said mixture until an association structure forms;
 (c) adding and mixing dry pigments until achieving a homogeneous
 mixture;
 (d) milling said mixture until uniform particle size is achieved; and
 (e) adding and mixing the mixture of (c) to the remaining lipstick
10 ingredients until a homogeneous mixture is achieved.

16. A lipstick composition comprising:

- (a) from 0.1% to 30% polar solvent;
 (b) from 5% to 20% of a surfactant mixture consisting essentially of
15 lecithin, PG-3 diisosterate, sorbitan oleate, cholesterol 12
 hydroxystegrate, and dipentaerythritol fatty acid ester.

INTERNATIONAL SEARCH REPORT

International Application No

PCT/US 93/08684

A. CLASSIFICATION OF SUBJECT MATTER
IPC 5 A61K7/00 A61K7/027

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 5 A61K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	EP,A,0 474 270 (UNILEVER NV) 11 March 1992 see the whole document	1
A	---	16
Y	PATENT ABSTRACTS OF JAPAN vol. 013, no. 562 (C-665) 13 December 1989 & JP,A,01 233 206 (KAO CORPORATION) 19 September 1989 see abstract	1
P,Y	EP,A,0 534 823 (L'OREAL) 31 March 1993 see page 3 see page 5 see page 6 see page 10 - page 11 ---	1
	-/--	

☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

* Special categories of cited documents :

- "A" document defining the general state of the art which is not considered to be of particular relevance
- "E" earlier document but published on or after the international filing date
- "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- "O" document referring to an oral disclosure, use, exhibition or other means
- "P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search

1 December 1993

Date of mailing of the international search report

22.12.93

Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2
NL - 2280 HV Rijswijk
Tel. (+ 31-70) 340-2040, Tx. 31 651 epo nl,
Fax: (+ 31-70) 340-3016

Authorized officer

Gac, G

INTERNATIONAL SEARCH REPORT

Int. Application No

PCT/US 93/08684

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	EP,A,0 217 105 (KAO CORPORATION) 8 April 1987 see the whole document ---	1,2,4,7, 8,16
A	CA,A,1 008 876 (HUTCHINSON ET AL.) 19 April 1977 see page 3 see page 6 see page 8 - page 14 ---	1,5,6, 10-12,16
A	EP,A,0 451 949 (UNILEVER PLC) 16 October 1991 see the whole document ---	1,2,6,7
A	PATENT ABSTRACTS OF JAPAN vol. 016, no. 356 (C-969)31 July 1992 & JP,A,04 108 718 (MIKIMOTO SEIYAKU KK) 9 April 1992 see abstract ---	1,16
A	SEIFEN, OLE , FETTE, WACHSE vol. 116, no. 7 , 24 April 1990 , AUGSBOURG pages 251 - 257 XP000165339 MEIJER H. 'Tenside und Wasser : Wesentliche Bestandteile kosmetischer Produkte und Anwendung' -----	1-4

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/US 93/08684

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
EP-A-0474270	11-03-92	US-A- 5085856 AU-A- 8118791	04-02-92 30-01-92
EP-A-0534823	31-03-93	FR-A- 2681781	02-04-93
EP-A-0217105	08-04-87	JP-B- 1053845 JP-C- 1668918 JP-A- 62053910 JP-C- 1703211 JP-B- 3071475 JP-A- 62096585 DE-A- 3683113 US-A- 4767625	15-11-89 29-05-92 09-03-87 14-10-92 13-11-91 06-05-87 06-02-92 30-08-88
CA-A-1008876	19-04-77	US-A- 3773021	20-11-73
EP-A-0451949	16-10-91	AU-B- 626749 AU-A- 7288391 US-A- 5120541	06-08-92 12-09-91 09-06-92